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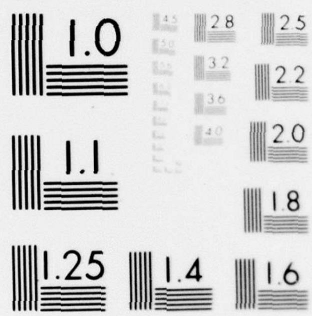
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DEUTERATED FLUIDS

DEUTERATED SYNTHETIC HYDROCARBON FLUID AND GREASE

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The replacement of hydrogen atoms with deuterium atoms in a synthetic hydrocarbon lubricant has been shown to substantially improve its oxidation resistance via the deuterium isotope effect. In addition, it was determined that the anti-friction bearing performance life of a MIL-G-81322 grease, formulated with the deuterium substituted fluid, increased significantly at 400°F (478°K). The results of this investigation are presented in this final report along with recommendations for the utilization of the deuterium isotope effect in developing a super clean instrument bearing grease.

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SUMMARY

INTRODUCTION

This final report is based on work performed under Independent Research Task No. ZR02206, Work Unit No. GC101, "Deuteration of Fluids for Naval Aircraft." The objective of this task is to provide scientific guidelines for the next generation of naval aircraft fluids based on deuterium modified molecules. A more complete definition of deuteration processing will define directional information for new approaches to wide temperature range fluids and greases.

High performance aircraft, instrumentation and associated machinery are often required to operate under extreme conditions of temperature and load and require longer life and more stable lubricants. At present, synthetic fluid materials have replaced petroleum based lubricants in a number of important aircraft applications and are finding increased usage based on better stability at higher temperature and improved viscosity index. In order to optimize the usefulness of these synthetic fluids, it was considered that a further modification of the chemical structure can provide superior chemical properties not available in currently used synthetic fluids.

Common to most lubricating fluids is the abundance of covalent hydrogen bonds. It is well established in the chemical literature that the replacement of hydrogen with deuterium (heavy hydrogen) will provide bond characteristics substantially stronger than ordinary hydrogen. Thus the deuterium modified synthetic fluid could exhibit increased stability at elevated temperatures and load due to stronger covalent bond formation.

The initial phase of this work was concerned with the deuterium modification of a synthetic hydrocarbon fluid and the investigation of its physical and chemical property changes related to lubricant performance characteristics. Grease formulations were then prepared from the deuterium substituted base fluid and investigated as a lubricant source for anti-friction ball bearings.

RESULTS

1. The direct exchange of hydrogen atoms with deuterium atoms in a synthetic hydrocarbon fluid (alpha-olefin oligomer) has been shown to be readily achievable even for high (97 atom percent) concentrations of deuterium. In performing the deuterium exchange, the original fluid does not undergo any structural isomerization or cracking, thus, the deuterated equivalent of the starting material is obtained.

2. The oxidation resistance of the synthetic hydrocarbon fluid increases as the deuterium concentration increases.

3. The specific gravity of the synthetic hydrocarbon fluid increases linearly as a function of deuterium content according to the following equation:

$$S.G. = 1.270 \times 10^{-3}\sigma + 0.8264 \qquad (1)$$

where:

S.G. = Specific gravity at room temperature

σ = Atom percent deuterated

4. When formulated into a MIL-G-81322 type grease, bearing performance life tests at 400°F (478°K) showed no increase in life at a 51 atom percent deuterium concentration when compared to the non-deuterated grease. Beyond this level modest increases in performance life were obtained at 73 and 88 atom percent deuterium content. The most striking increase in performance life occurs at deuterium concentrations of 94 atom percent and higher with a greater than five-fold increase occurring at 97 atom percent deuterium.

5. A 2.5-fold increase in bearing performance life was achieved in the neighborhood of 50 atom percent deuterium when equal volumes of 97 atom percent deuterium and nondeuterated greases were intimately blended. On the contrary, the oxidation resistance of the corresponding fluid blend was not as good as that observed for the 51 atom percent deuterated fluid.

6. A sample of synthetic hydrocarbon fluid which was treated with hydrogen instead of deuterium showed some improvement in oxidation resistance over the non-treated fluid but bearing performance tests showed no increase in life.

CONCLUSIONS

The deuterium "Isotope Effect" of C-D vs. C-H bonds has been shown to significantly improve the anti-friction bearing performance of a synthetic hydrocarbon grease via improved oxidation resistance. The degree of improvement has been found to be dependent not only on the deuterium content, but also on the manner in which the deuterium atoms are distributed throughout the molecules.

In spite of the high cost involved in material and processing of deuterium substituted synthetic hydrocarbon fluid, the result of this modification is considered to be potentially cost effective. The limited life of many aircraft instruments is directly related to bearing lubricant degradation and deterioration. Typically, instrument bearings often require sparse lubrication to provide low and more stable torque, less noise, jog-free operation, etc. As a result of the minute quantities required, lubricant cost becomes almost insignificant, even at 500 dollars per pound, when considering extended life versus cost of maintenance and/or replacement of bearing or components. It is interesting to note that one pound of grease will theoretically lubricate 65,000 typical R-3 spin motor bearings. This would correspond to about 0.8 cents per bearing.

RECOMMENDATIONS

The synthetic hydrocarbon fluid utilized for deuterium processing is the base oil now used in Military Specification MIL-G-81322 which is a widely used multipurpose (wheel bearings to instrument bearings) grease. Although its performance in instrument bearings has resulted in the replacement of many other previously used greases, the MIL-G-81322 grease lacks the extreme cleanliness required for super precision miniature bearings. It is, therefore, recommended that a development program be established for providing a "tailored" superclean instrument bearing oil and grease based on deuterium modified synthetic hydrocarbon fluid.

In addition, investigations into the effect of unsaturation and deuterium atom location on oxidation resistance and bearing performance life of model chemical species would provide further insights into the mechanism involved in the utilization of the deuterium isotope in lubrication.

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B A C K G R O U N D

Details concerning initial investigations into the effect of deuterium exchange on a synthetic hydrocarbon lubricant have previously been reported (1,2,3). The substitution of a hydrogen atom with a deuterium atom in a chemical bond such as C-H will significantly reduce reaction rate constants in corresponding chemical reactions if cleavage of the particular chemical bond under consideration occurs in the rate determining or slow step in the reaction sequence. This characteristic is generally referred to as the "isotope effect" and is most prominent with chemical bonds containing hydrogen and isotopes thereof. The theoretical basis and explanation of the "isotope effect" due to nuclear mass differences has been adequately covered in the literature. (4)

The oxidation of hydrocarbon molecules has been shown (5) to proceed through a free radical mechanism with the rate determining step being the formation of hydroperoxides (ROOH) via peroxide free radical (ROO·) attack on the hydrocarbon substrate (RH). Since cleavage of a C-H bond is required in the rate determining step, the substitution of hydrogen atoms with deuterium atoms will significantly slow down the oxidation process.

The deuterium "isotope effect" for enhanced oxidation resistance was investigated using a synthetic hydrocarbon grease in an anti-friction bearing performance life test (Figure 1). No increase in bearing performance life over the nondeuterated grease was observed at a 51 atom percent deuterium concentration. Beyond this level modest increases in performance life were observed at 73 and 88 atom percent deuterium concentrations. The most striking responses occur at deuterium concentrations of 94 atom percent and higher with a greater than five-fold increase occurring at 97 atom percent deuterium concentration. Oxidation studies on the partially deuterated fluids indicated that even at the 51 atom percent deuterium level, significant improvements in oxidation resistance are obtained (Table IV) yet the bearing performance results showed no improvement in life. It was therefore of interest to further investigate the significance of these findings.

E X P E R I M E N T A L

The exchange of hydrogen in the synthetic hydrocarbon fluid with deuterium was performed via a process which is proprietary in nature and differs in some respects to the laboratory preparation detailed in reference (6).

The synthetic hydrocarbon fluid used is designated RL-714 and contained no additives. This fluid has an average molecular weight of 515.

Grease samples were formulated under laboratory conditions and contained additives required to meet the requirements of Military Specification MIL-G-81322. The difference in specific gravity between the nondeuterated and deuterated synthetic hydrocarbon fluids was taken into account when formulating.

Bearing performance tests were conducted in accordance with Federal Test Method Standard No. 791B Method 333.

Bomb oxidation tests on fluids were performed in accordance with ASTM Method D-942 for grease samples. Two grams of fluid were substituted for the four grams of grease required in this method.

The specific gravity of fluids was determined using a Weld type pycnometer.

The Bromine Number was determined in accordance with ASTM Method D-1159.

The Nuclear Magnetic Resonance Analysis was obtained on a 60 MHz Perkin-Elmer R-24 spectrometer.

The thermogravimetric analysis was performed on a DuPont 950 TGA module.

RESULTS

MASS SPECTRAL ANALYSIS

The increased bearing performance life for the highly deuterated synthetic hydrocarbon grease shown in Figure 1 is considered to be primarily due to the greater oxidation resistance of the C-D bond vs. the C-H bond. In order to explain the lack of response at the 51 atom percent deuterium level in spite of its improved oxidation resistance, it was postulated that the increase in bearing performance life is dependent on the amount of completely or nearly completely deuterated synthetic hydrocarbon oligomers present. From a probabilistic standpoint, the 97 atom percent deuterium fluid should possess a number of molecular units which are completely deuterated. If the average oligomer is a $C_{36}H_{74}$ molecular unit then the 97 atom percent deuterium level average molecular formula would correspond to $C_{36}D_{72}H_2$, thus molecular units of $C_{36}D_{74}$ and $C_{36}D_{70}H_4$ are theoretically and probabilistically possible. In contrast, at the 51 atom percent deuterium level the average molecular unit would correspond to $C_{36}D_{38}H_{36}$. Significant concentrations of $C_{36}D_{74}$ and $C_{36}D_2H_{72}$ would then be highly unlikely.

In order to experimentally verify this hypothesis, mass spectral analysis was performed on the 51 and 97 atom percent deuterium fluids as well as the nondeuterated fluid. The mass spectra of the nondeuterated fluid indicated considerable fragmentation which is characteristic of long-chain hydrocarbons. The principal peaks appear at masses below m/e equal to 100. The predominant peaks in the nondeuterated fluid are closer in relative abundance to those of normal long-chain hydrocarbons than to those of hydrocarbons heavily branched with short-chain branches. Peaks with m/e equal to 29 and 43 are especially abundant. It appears that the nondeuterated fluid consists of a long-chain backbone with several moderately long side chains.

The mass spectra of the 97 atom percent deuterium fluid shows a high proportion of $C_nD_{2n+1}^+$ ions as indicated by peaks with m/e equal to 34, 50, 66, 82, 98, 114, 130 and 146. Allowing for contributions of $^{13}C\ C_{n-1}D_{2n}^+$ to the various peaks, the ratio of peak intensities for $C_nD_{2n}H^+$ to C_nD_{2n+1} has been calculated for various C_n fragments. For example the ratio

$C_5D_{10}H^+$ to $C_5D_{11}^+$ has been found to be equal to 0.16 indicating that 16 percent of the C_5 fragments have a single hydrogen atom. This corresponds to an atom percent deuterium concentration of 98.5 for this particular fragment. Likewise the $C_3D_6H^+$ to $C_3D_7^+$ ratio has been found to be equal to 0.11 again corresponding to approximately 98.5 atom percent deuterium concentration for the C_3 fragments.

The mass spectrum obtained on the 51 atom percent deuterium fluid showed a rather random deuterium distribution since there were significant peaks for all degrees of deuterium at each C_n fragment with middle m/e values having the highest abundance as expected from probabilistic considerations.

Although it was not possible to determine the actual degree of completely or nearly completely deuterated molecules in each sample because of lack of parent peak information (high fragmentation), the spectra are consistent with the proposed hypothesis for explaining the lack of response in bearing performance at the 51 atom percent deuterium level.

BEARING PERFORMANCE LIFE OF ADMIXED GREASE

If the above hypothesis is indeed operative then one would expect to observe an increase in bearing performance life in the neighborhood of 50 atom percent deuteration if a high degree of completely deuterated or nearly complete deuterated molecules are present in a grease formulation. An approach toward providing such a grease is simply to admix two greases, one of which is highly deuterated, the other being completely nondeuterated. For example, if equal volumes of 100 atom percent deuterium grease and nondeuterated grease are mixed, the resultant grease would have a deuterium concentration of 50 atom percent. The 50 atom percent deuterium grease differs from the 50 atom percent deuterium admixed grease in the molecular distribution of deuterium atoms. The unblended grease has deuterium atoms distributed over every molecule while in the admixed grease 50 percent of the molecules are highly deuterated with the balance containing only hydrogen atoms.

An admixed grease containing 48.5 atom percent deuterium was prepared and bearing performance life tests were performed. The result obtained is shown in Figure 1 along with comparison data for the unblended greases. It can be observed that the ratio of bearing performance life for the 48.5 atom percent deuterium admixed grease vs. the nondeuterated is 2.5 times greater than the ratio of bearing performance life for the 51 atom deuterium grease vs. the nondeuterated grease. Thus, greases with similar deuterium contents have been found to exhibit marked differences in bearing performance life. This result lends support to the hypothesis that the bearing performance life of deuterated greases is also a function of the degree of completely or nearly complete deuterated molecules present in the synthetic hydrocarbon oligomer.

BROMINE NUMBER

Another area of investigation centered on establishing the degree of unsaturation present in the various base fluids. It is generally known that unsaturation will decrease the oxidation resistance of lubricating oils. A

common method used to determine the degree of unsaturation is known as the Bromine Number. Essentially, bromine will add to carbon-carbon double bonds to form brominated compounds. By determining the amount of bromine reacted in a sample compound a measure of the degree of unsaturation present can be obtained. Table I lists the bromine numbers obtained on the various fluids under study. Normally bromine numbers are expressed as grams of bromine atoms reacted per 100 grams of sample. Because of density differences between the various deuterated samples, the bromine numbers were also calculated based on an equal volume relationship. These figures can then be used for better comparison purposes. Also, an estimate of the number of double bonds per molecule was calculated based on estimated molecular weights. Diisobutylene was used as a standard to determine the accuracy of the procedure. Because of the relatively low values obtained the question arose as to whether the bromine number procedure was indeed indicative of the amount of unsaturation present or was there a possibility that side reactions were contributing to the bromine number determinations. Indeed it was learned (7) that tertiary hydrogen atoms could be displaced by bromine atoms since these atoms form weaker bonds than secondary or primary hydrogen atoms. In the diisobutylene standard which was used there are no tertiary hydrogen atoms, therefore, the experimental value of the bromine number is in good agreement with the theoretical value. An alternative method is required in order to establish whether small amounts of unsaturation are present in the synthetic hydrocarbon-deuterocarbon samples.

NUCLEAR MAGNETIC RESONANCE ANALYSIS

In addition to the determination of the deuterium concentration in each sample by NMR, information was also obtained on the ratio of methylene protons to methyl protons remaining as a function of deuterium content. Table II shows the results obtained and also calculated values for the ratio of methylene groups vs. methyl groups. As can be observed, this ratio remains essentially constant in going from the non-deuterated fluid to the highly deuterated fluid. Apparently the energetics of the deuteration process is enough to compensate for the differences in reactivity between secondary and primary hydrogen positions. As deuteration proceeds methylene hydrogens are being replaced at the same rate as methyl hydrogens thus leading to the constant ratios which were observed.

GREASE PROPERTIES

Table III lists some typical properties of both non-deuterated synthetic hydrocarbon grease and 97 atom percent deuterated grease. This table shows that the other desirable properties of the deuterated grease are essentially equivalent to the non-deuterated grease even though oxidation resistance and bearing performance life have been dramatically improved. These results point to the unique characteristics obtained in utilizing the deuterium kinetic isotope effect in lubrication. Essentially, significant increases in bond strength have been achieved without drastically altering the chemical nature of the base fluid. For example, the bond strength and subsequent oxidation resistance could have been improved by substituting methyl groups for hydrogen atoms in the synthetic hydrocarbon fluid but the resulting fluid would then have possessed properties quite different from the original fluid.

OXIDATION RESISTANCE

Table IV shows the results of three different analytical procedures used to investigate the oxidation characteristics of various deuterated and non-deuterated fluids. It can be observed that as the deuterium content is increased the oxidation resistance of the fluid also increases. When equal volumes of non-deuterated and 97 atom percent deuterated fluid were mixed to give an effective deuterium concentration of 48.5 atom percent, the oxidation resistance of this admixed fluid was found to be appreciably less than the 51 atom percent deuterated fluid. This was somewhat unexpected since the corresponding grease formulations exhibited an opposite trend in the bearing performance life evaluation.

A sample of original non-deuterated fluid, which had been processed in the same manner as the deuterated fluids with the exception that hydrogen gas was used instead of deuterium, exhibited somewhat of an increase in oxidation resistance yet its bearing performance life showed no improvement over the unprocessed non-deuterated fluid.

THIN FILM STABILITY TESTS

Thin film stability comparison tests were conducted on samples of non-deuterated and 97 atom percent deuterated oils, with and without oxidation inhibitors. A sample of formulated MIL-L-81846 Lubrication Instrument Oil was included as a control sample. The test consists of transferring a 0.35 ± 0.05 gram sample to a stainless steel planchet (5 cm diameter and 1 cm deep). The planchets containing the oil are held for 6½ hours in a gravity convection oven maintained at $350 \pm 4^{o}F$ ($450 \pm 2^{o}K$). The weight loss, flow properties and final appearance of the oil were noted. Final flow properties were evaluated by tilting the planchets at approximately a 60^{o} angle and determining the amount of residual oil flowing into a small cup after 5 minutes.

The results of weight loss and weight of oil flowing from the planchets after the tests are shown in Table V.

The most noticeable difference among all the samples tested was the heavy lacquer formation and lack of oil flow for non-deuterated oil versus the 97 atom percent deuterated oil. These two oils with antioxidants compare quite favorable with the MIL-L-81846 instrument oil.

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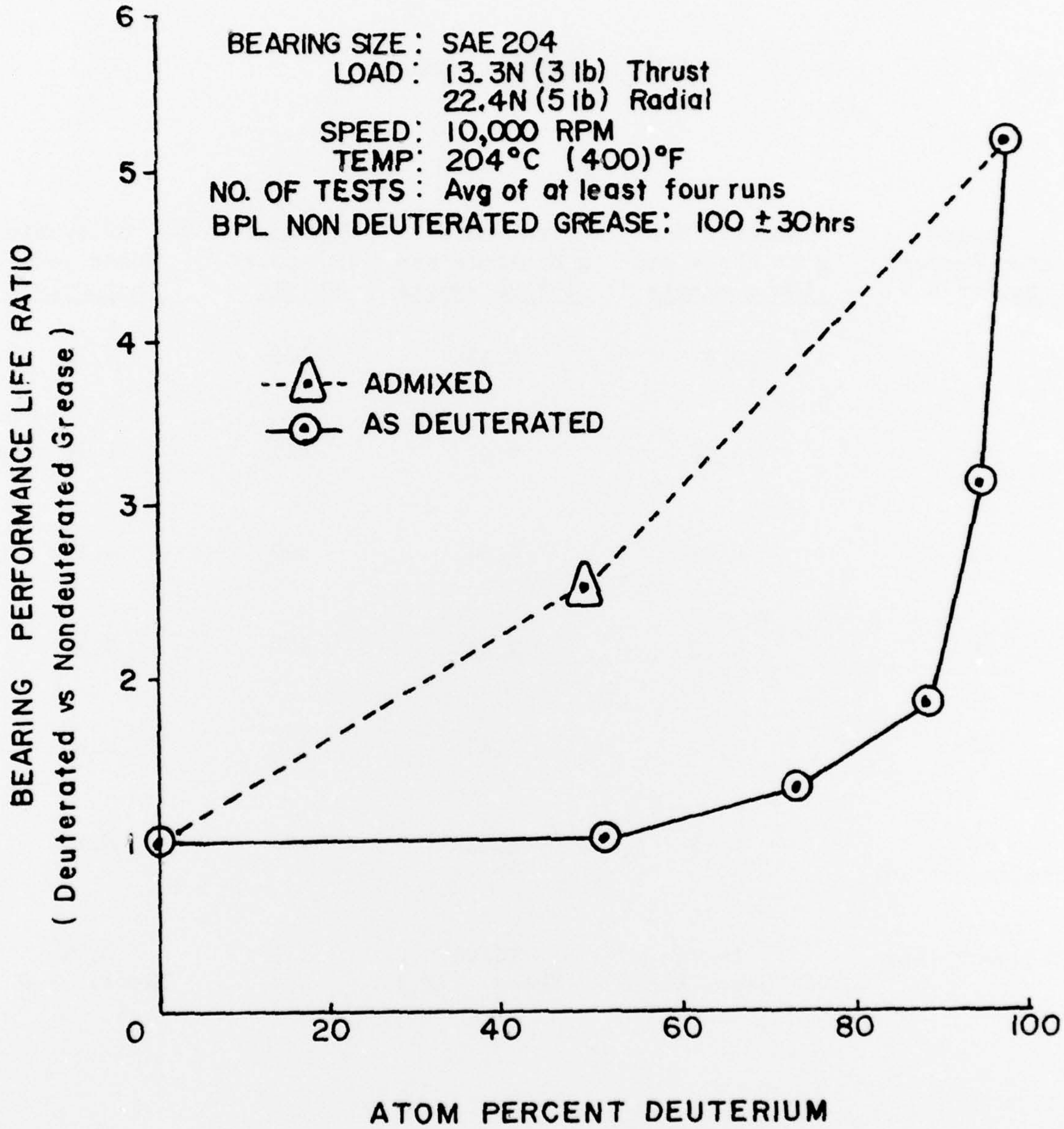

Figure 1. Anti-friction Bearing Performance Life Ratios of SH Greases

TABLE I. BROMINE NUMBER

Fluid Atom Percent Deuterated	Bromine No. g Br Atoms per 100 g sample	Bromine No. g Br Atoms per 100 cc sample	Estimated Molecular Weight	No. of Double Bonds per Molecule
0	10.8	8.93	506	0.34
51	8.59	7.67	542	0.29
73	4.74	4.34	560	0.17
88	2.70	2.53	571	0.10
97	1.50	1.43	578	0.05
0 (Hydrogenated)	4.15	3.43	506	0.13
Diisobutylene	141.6 Theor. 142.6	101.9 Theor. 102.6	112 112	0.99 Theor. 1.0

TABLE II. NMR ANALYSIS

Atom Percent Deuterium	methylene H / methyl H	$\frac{CH_2}{CH_3}$
0	3.107	4.826
51	3.158	4.737
73	3.192	4.788
88	3.123	4.685
97	*	*

* Absorption too small for accurate determination

TABLE III. PROPERTIES OF DEUTERATED AND NON-DEUTERATED GREASES

Property	Non-Deuterated	97 Atom Percent Deuterated
Dropping Point, F (K)	568 (571)	533 (551)
Worked Penetration	312	317
Oil Separation 350F (450K) %	4.3	5.9
Load Wear Index	43.5	41.2
Steel-On-Steel Wear (1200 RPM, 40 Kg Load)		
52100 167F (348K) mm	0.50	0.45
M-10 400F (478K) mm	0.59	0.40
Low Temp. Torque, -65F (219K)		
Starting g-cm	7,699	6,933
Running g-cm	929	1,136
Evaporation, 350F (450K) %	7.33	6.43

TABLE IV. OXIDATION RESISTANCE

Atom Percent Deuterium	A Pressure Loss PSI (kPa)	B Intensity Counts/sec	C Wt. Loss %
0	96.5 (665)	3400	70.0
51	4.5 (31)	300	-
73	2.5 (17)	-	-
88	0 (0)	-	-
97	0 (0)	Est. 25	27.6
48.5 (Admixed)	23.5 (162)	1500	-
0 (Hydrogenated)	56 (386)	-	-

A = Bomb Oxidation Test ASTM D942 2 g fluids substituted for 4 g grease, 168 h, 210F (372K)-Bombs charged to 110 PSI (758 kPa)

B = Chemiluminescence Test 266F (403K), Counts per sec at 6000s in air/oxygen atmosphere.

C = Thermogravimetric analysis wt. loss percent at 572F (573K) in oxygen atmosphere.

TABLE V. THIN FILM STABILITY

	Non Deuterated	97 Atom Percent Deuterated
Wt. of oil tested (grams)	0.3533	0.3514
Wt. loss of oil (grams)	0.1459	0.1568
% loss	41.3	44.6
Wt. of oil flow (grams)	0.000	0.1003
% oil flow of remaining oil	0.0	49.0
Particulate matter	None	None
Lacquer	Heavy	Faint

	Non Deuterated with Anti-Oxidant	97 Atom Percent Deuterated with Anti-Oxidant
Wt. of oil tested (grams)	0.3510	0.3492
Wt. loss of oil (grams)	0.0333	0.0274
% loss	9.5	7.8
Wt. of oil flow (grams)	0.2823	0.2802
% flow of remaining oil	88.9	87.1
Particulate matter	None	None
Lacquer	None	None

	MIL-L-81846
Wt. of oil tested (grams)	0.3539
Wt. loss of oil (grams)	0.0577
% loss	16.3
Wt. of oil flow (grams)	0.1980
% oil flow of remaining oil	66.8
Particulate matter	None
Lacquer	None

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